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Pulsed Microwave Irradiation of Graphite/Epoxy Composites

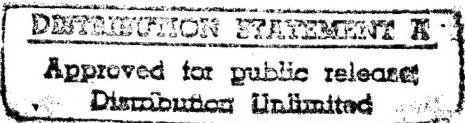
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**PULSED MICROWAVE IRRADIATION OF
GRAPHITE/EPOXY COMPOSITES**

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ABSTRACT

We have measured the microwave-induced damage to the near-surface region of a graphite/epoxy composite material for $1.1\text{-}\mu\text{s}$ pulses at a frequency of 2.865 GHz and a pulse power of up to 8 MW. Rectangular samples were irradiated by single-pass TE₁₀ traveling wave pulses inside a WR-284 waveguide, and in situ and post irradiation studies were performed to characterize the material modifications induced by the microwave pulses. The results of time-resolved optical measurements in vacuo show that surface decomposition of the epoxy resin occurs for incident pulse powers exceeding 1.1 MW, and that the surface damage is accompanied by a large increase in the reflected microwave power. Simultaneous with the onset of surface decomposition, we observe significant light emission from the sample and a large enhancement of the gas pressure in the test cell. The large increments in the reflected power and light emission are attributed to the formation of a plasma due to electrical breakdown of the gas at (or near) the sample surface.

INTRODUCTION

Graphite/epoxy composite materials are rapidly replacing metals, especially in applications where high strength, weight and dimensional stability are important. Furthermore, it is common practice to design and produce graphite/resin composites that optimize particular selected properties, such as tensile, compressive and flexural strengths. The reinforcing graphite filaments in the composite are used to obtain the desired mechanical properties, and the epoxy is used to yield a finished part with stable, high strength laminates and low void contents. In this paper a study of the energy deposition and material damage of a graphite/epoxy composite due to excitation by high-power pulsed microwave radiation is presented. The primary goals are to determine the damage threshold as a function of the excitation conditions and to use the data in the design and materials selection of radiation-resistant composites.

EXPERIMENT

A high-power pulsed klystron was used to generate microwave pulses having a frequency of 2.856 GHz and a maximum peak power of 10 MW. By gating the rf drive, the pulse duration was varied between 135 ns and 2 μ s with a risetime of approximately 70 ns. A schematic diagram of the experimental setup is shown in Fig. 1. The output of a highly stable cw master oscillator is gated by a PIN diode that provides a variable-length 500-mW drive pulse to a driver klystron, which in turn produces a 135-2000 ns drive pulse for the high-power klystron. The output pulse from the klystron passes through a high-power cir-

culator, which then feeds into a WR-284 copper waveguide. The waveguide is terminated with an impedance-matched, water-cooled load to minimize reflections and ensure that the sample is irradiated by a single-pass, TE_{10} traveling-wave pulse.

The test cell consists of a copper waveguide section that allows insertion of a sample into the microwave field without requiring the venting of the waveguide system. The samples are inserted into the test cell with a linear motion feedthrough and are suspended in the center of the WR-284 waveguide by an alumina holder. An E-H tuner is used in the test section to reduce the impedance mismatch associated with the presence of the sample in the waveguide cavity. Using a cw low-power microwave source, the tuner is adjusted to minimize the reflected signal when the sample is inserted into the waveguide. The test cell was evacuated to a pressure of about 4×10^{-7} torr, and the waveguide assembly, not including the test cell, was pressurized to 30 psig with Freon-12.

In this paper values of the incident microwave power are quoted rather than the power density, because the presence of the sample may cause some local modification of the field of the TE_{10} traveling wave pulse. If one assumes that the presence of the sample does not significantly perturb the TE_{10} guided mode in the test cell, then the power density incident on the sample can be obtained by dividing the pulse power by 12.25 cm^2 .

The graphite/epoxy samples used in the experiment were Thorne 300 graphite filaments and Narmco 5208 epoxy, which is one of the most common graphite/epoxy composites. The samples had a unidirectional layup and a thickness of 3.18 mm. Each sample was cut to a size of 5x5 mm, so that the cross-sectional area of the sample ($\approx 0.25 \text{ cm}^2$) would be much smaller than the cross-sectional

area of the test cell ($\approx 24.5 \text{ cm}^2$). The Narmco 5208 epoxy was cured at a temperature of 450 K. The graphite filaments in the composite have a diameter of about $6 \mu\text{m}$ and comprise approximately 61 % of the sample volume. The electrical resistivity of the graphite is about $20 \mu\Omega\text{-cm}$, from which a calculated linear absorption coefficient of about 230 cm^{-1} is obtained for 2.856-GHz microwave radiation [1].

RESULTS AND DISCUSSION

Time-resolved measurements of the microwave reflectivity (R) from the graphite/epoxy samples were performed. Each sample was initially impedance-matched at low microwave powers (i.e., $\text{VSWR} < 1.05$), in order to minimize the reflected power. For incident microwave pulse powers exceeding about 1.1 MW, an abrupt increase in the microwave reflectivity was observed near the end of the $1.1\text{-}\mu\text{s}$ pulse. (When the sample was removed from the test cell, no change in R was observed over the same range of microwave powers.) For higher incident microwave pulses, the onset of the increased reflectivity occurred at earlier times during each pulse. Figure 2 shows the time required for the sudden increase in R as a function of the incident microwave power. Each data point was obtained by averaging 20 different shots at a fixed pulse power, and each error bar designates the standard deviation of the data from the different shots at a fixed ($\pm 5 \%$) incident pulse power.

The surface topography of the samples was studied with a scanning electron microscope (see Fig. 3). For incident microwave powers exceeding the threshold value ($\sim 1.1 \text{ MW}$) at which the high reflectivity is observed, the

samples show evidence for localized surface decomposition of the epoxy. Further increases in the pulse power beyond 1.1 MW cause more of the epoxy to be lost from the sample. After exposure to several shots at pulse powers above about 5 MW, only bare graphite filaments are visible on the surface. Figures 3b and 3c are SEM photographs of samples after excitation by twenty shots, each shot having an incident pulse power of 5.1 and 6.9 MW, respectively. Several graphite filaments are broken at pulse powers exceeding approximately 6 MW, which is most likely due the electrical discharge associated with the breakdown of the gas near the target surface [2].

The onset of depolymerization of the epoxy in a vacuum occurs at a temperature of about 600 K [3], whereas the melting point of graphite is approximately 4300 K [4]. Thus, the damage threshold for the epoxy is expected to be much lower than for the graphite filaments, which is consistent with the SEM results shown in Fig. 3.

A video camera was used to monitor each sample before and after excitation by the high-power microwave pulses. We found that the microwave irradiation caused the emission of visible light from the sample surface. When the sample and holder were removed from the waveguide, no light emission was detected from the empty waveguide. A Hamamatsu R2055 photomultiplier tube was employed to measure the light emission as a function of the incident microwave power. The results for the maximum photomultiplier output are displayed in Fig. 4 for 1.1- μ s pulses and a photocathode voltage of 900 V. The light emission incident on the photocathode was attenuated by a 10%-transmission broadband neutral density filter and two fused silica windows. Each data point in the figure represents the average of twenty different shots at a fixed incident pulse power, and each error bar designates the standard deviation of the

data. The threshold for the light emission occurs at about 1 MW, which is approximately equal to the threshold for the sudden increase in the reflected microwave power.

The pressure of the gas in the test cell was continuously monitored by an ion gauge, which was located about 40 cm downstream from the sample. Figure 5 shows the maximum increase in the gas pressure as measured at the ion gauge for several different pulse powers. Each data point designates the average increase resulting from ten different 1.1- μ s pulses. The initial base pressure of the gas at the ion gauge was in the range of $3\text{-}8 \times 10^{-7}$ torr.

Our interpretation of the experimental results is as follows: The small increase in the gas pressure at a pulse power near 1 MW is probably caused by microwave heating and a subsequent desorption of water, oxygen and other absorbed species from the sample. The electric field associated with the high-power microwave radiation accelerates the electrons and desorbed ionized species and causes further ionization via collisions with neutral particles. The additional electrons are also accelerated to high velocities by the pulse, leading to more charged species and subsequent electrical breakdown of the gas near the target surface.[4] The electrical discharge associated with the gas breakdown causes further heating and decomposition of the surface. The additional loss of material from the surface leads to much larger enhancements in the plasma density and gas pressure. The presence of the microwave-induced plasma is responsible for the abrupt increase in the reflectivity of the microwave pulse from the test cell, which reduces the duration of the exposure of the sample to the high-power microwave pulse. The emission of the visible light results from the relaxation of the plasma at (or near) the target surface.

SUMMARY AND CONCLUSIONS

Graphite/epoxy composite samples were irradiated in a waveguide by single-pass high-power microwave pulses, and post-irradiation studies show that the surfaces of the samples can be damaged by the microwaves. For pulses with a duration of 1.1- μ s, surface damage is apparent for incident microwave powers exceeding about 1.1 MW. The onset of surface damage is accompanied by a large increase in reflected microwave radiation and significant emission of light from the target area. Examination of the irradiated samples shows that the epoxy at the surface decomposes, whereas the graphite filaments appear primarily undamaged. We believe that the increase in reflected microwave power, emission of visible light from the target area, and surface damage are attributed to the formation of a plasma due to electrical breakdown of the gas at (or near) the sample surface.

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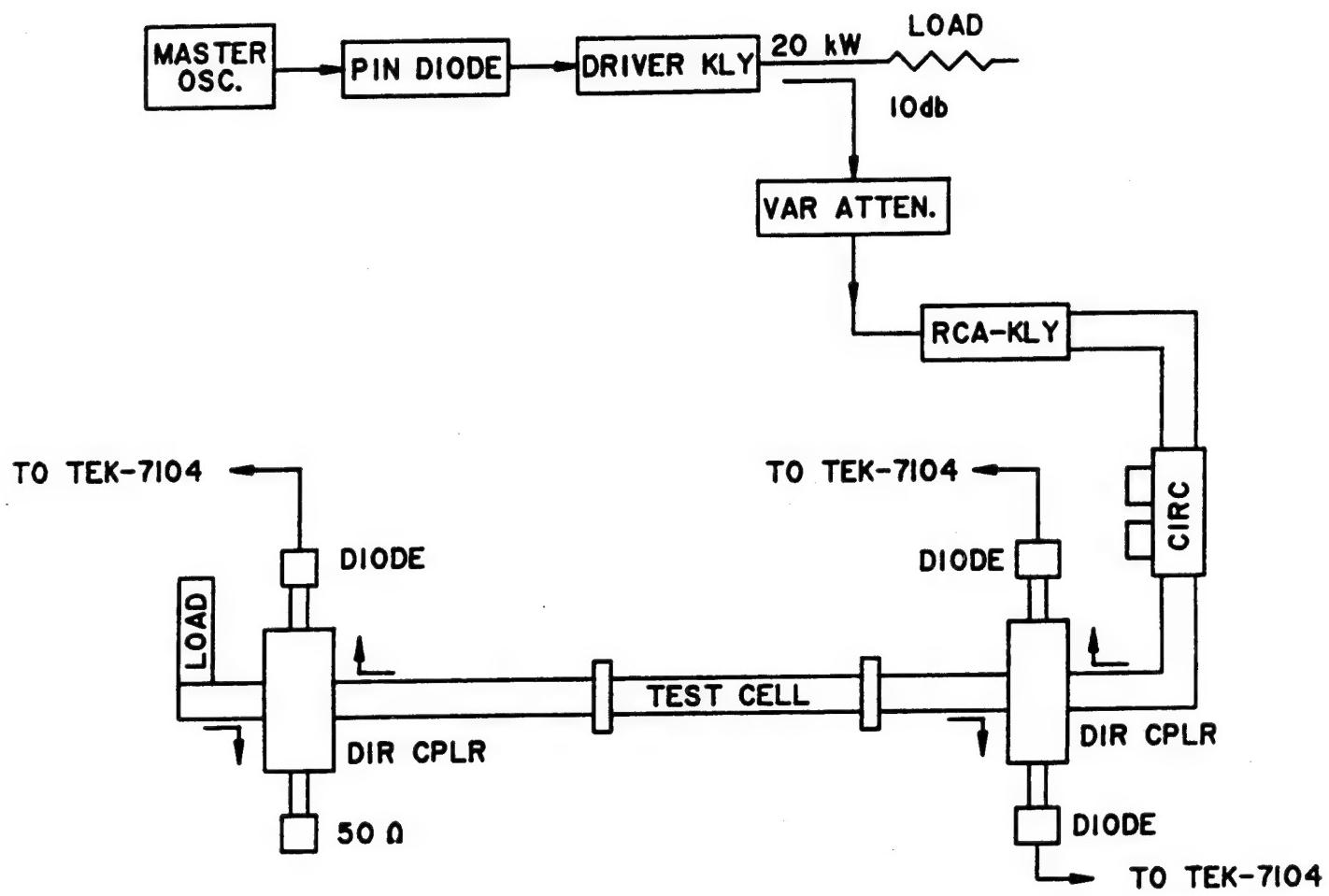


Fig. 1 Schematic diagram of the experimental setup.

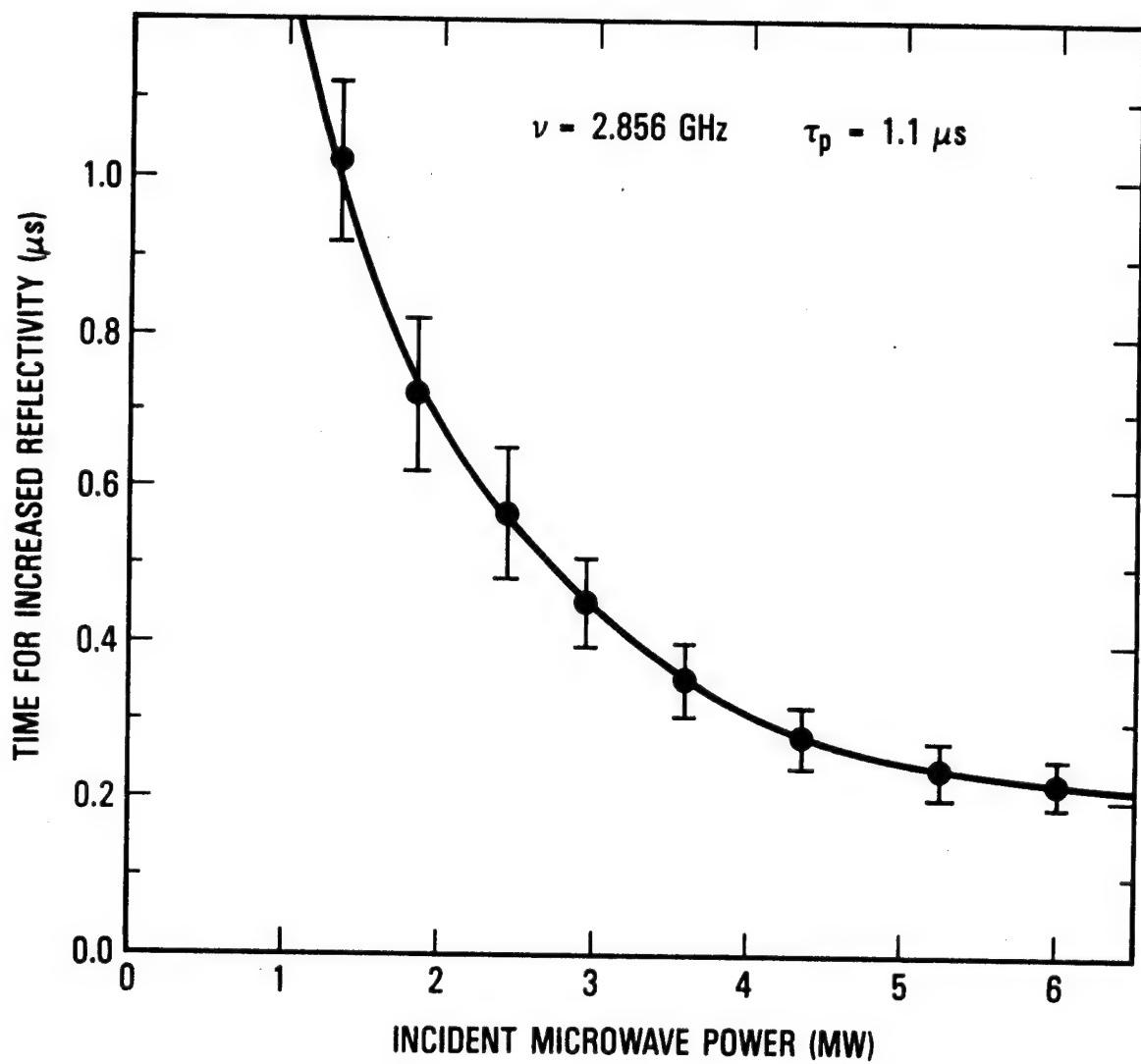


Fig. 2 Time required for the abrupt increase in the reflectivity of the microwave radiation as a function of the incident pulse power.

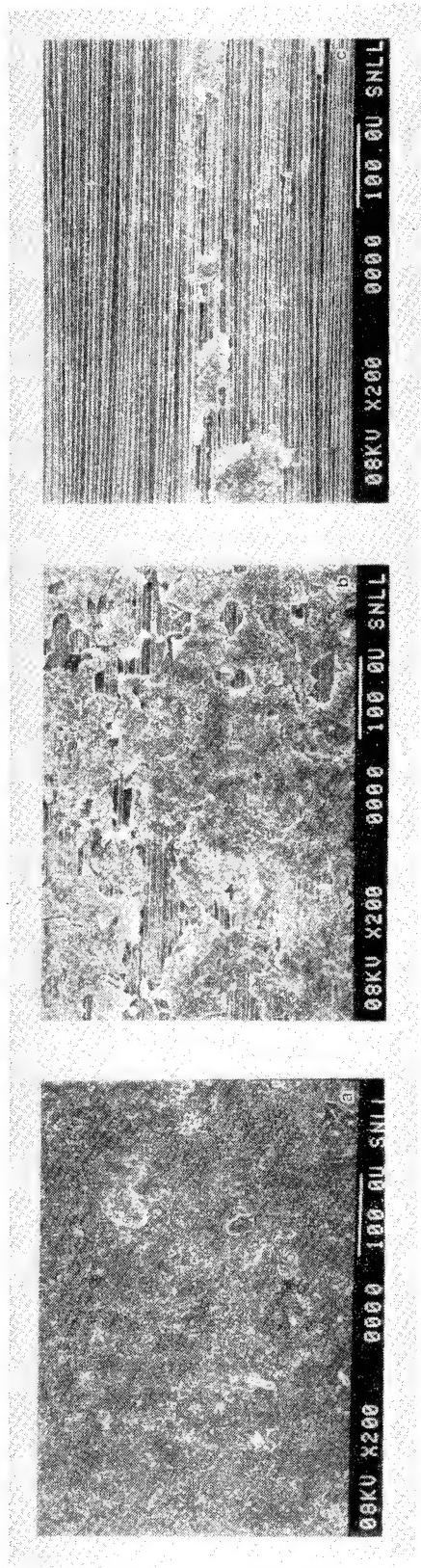


Fig. 3 SEM micrographs of the sample surface. Fig. 3(a) shows an unirradiated sample, 3(b) shows a sample after irradiation by twenty 1.1- μ s pulses at a power of 5.1 MW, and 3(c) shows a sample after irradiation by twenty 1.1- μ s pulses at a power of 6.9 MW. The magnifications and scale markers are shown at the bottom of each photograph.

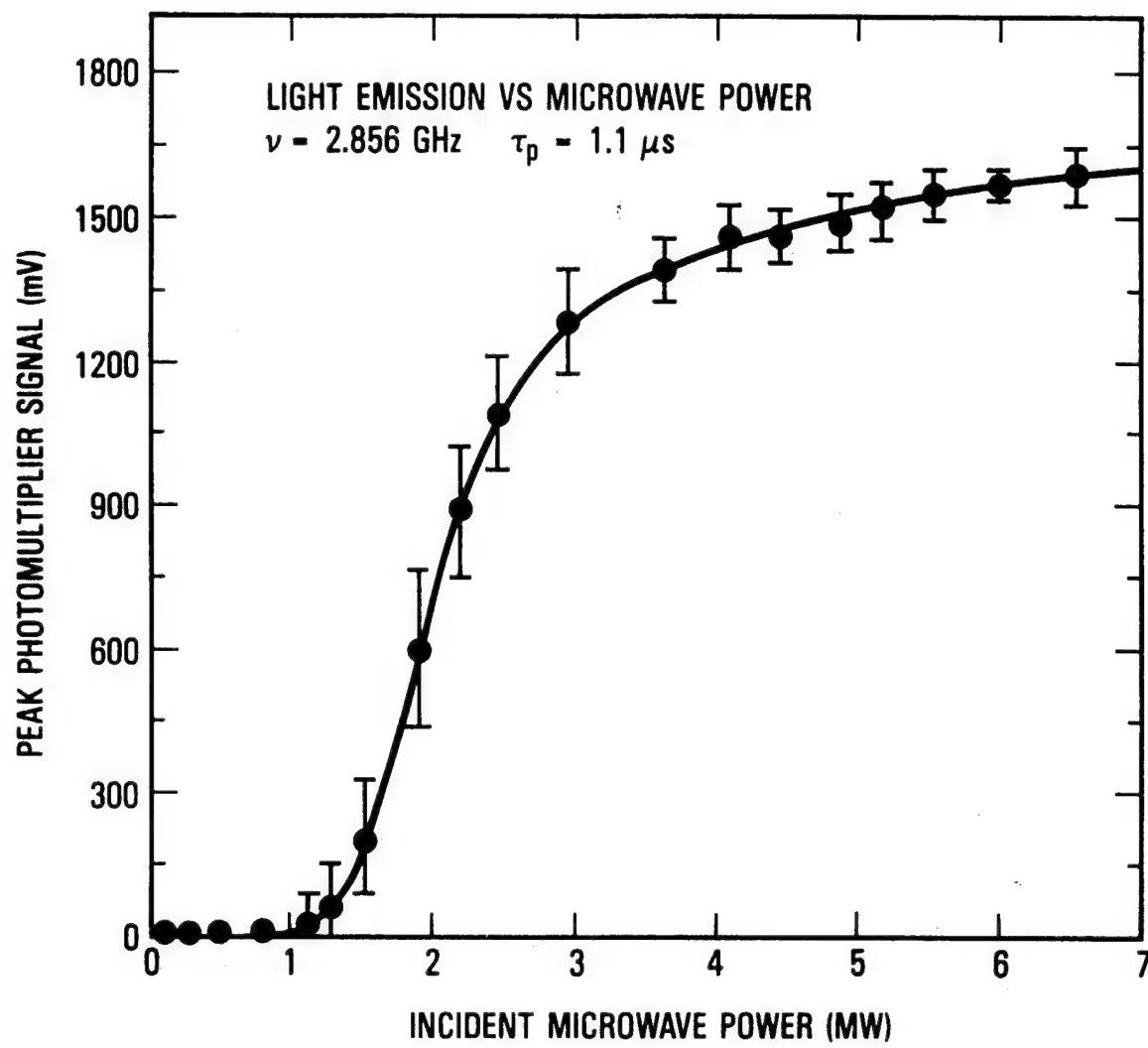


Fig. 4 Peak photomultiplier output as a function of the incident microwave power.

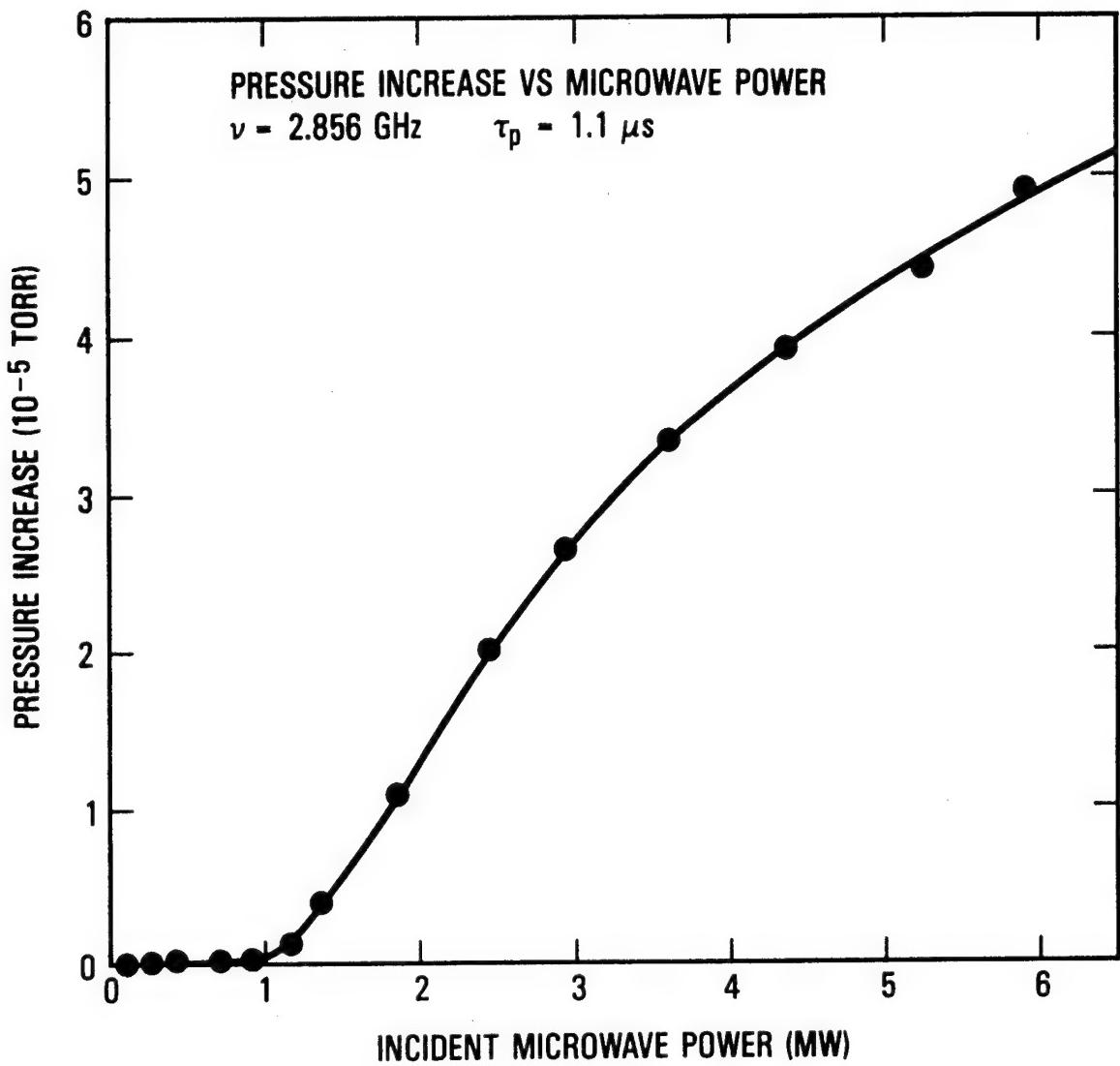


Fig. 5 Measured increase of the gas pressure in the test cell as a function of the incident microwave power.

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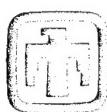
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